REMARKS

Reconsideration of the above-identified patent application, as amended, is respectfully requested. By means of the present Amendment, independent claims 12 and 24 have been amended, while claims 19 and 20 have been cancelled. Accordingly, claims 12-18 and 21-24 are now pending in the application. Of the foregoing, claims 12 and 24 are independent.

In the Office Action dated December 29, 2003, the Examiner rejected claims 12, 13, 19, and 24 under 35 U.S.C. 102(b) as being anticipated by JP 07-207,357 ("JP '357"). According to the Examiner, JP '357 teaches a device for filtering and adding grain refining material to a metal melt, the device comprising a first filter, a grain refining material feed downstream of the first filter, and a second filter downstream of the first filter, as set forth in claim 12. The Examiner also rejected the balance of the claims in the application under 35 USC 103(a) as being unpatentable over JP '357 in view of one or more of US 4,790,873 ("US '873"), US 4,113,241 ("US '241"), and US 4,790,870 ("US '870").

In response to these grounds for rejection, applicants have amended independent claims 12 and 24 to incorporate the limitations of claims 19 and 20. Consequently, all claims now specify that the second filter comprises a porous filter medium in the form of a deep-bed filter. It is submitted that claims 12 and 24 are neither anticipated nor rendered obvious by the prior art of record.

In particular, submitted herewith is a copy of JP '357 in Japanese as well as in unsworn English translation thereof. As can be seen, JP '357 discloses a device for filtering and adding a grain-refining material to a metal melt, said device comprising a first filter and a second filter, wherein the grain-refining material feed is disposed between the first filter and the second filter. See the abstract and claim 1 of JP '357. JP '357 further teaches that the second filter should be in the form of disposable plate filters, rather than in the form of an expensive deep-bed filter as now specified in claims 12 and 24. Thus, at para. [0013], JP '357 states that adding grain-refining material leads to an accelerated clogging of the filter in the second filtering stage due to the formation of coarse Al-Ti and Al-Ti-B impurities of the grain refining material. Also, at para. [0016] and in claim 4 of JP '357, the second filter is disclosed as being in the form of a "cheap and therefore disposable plate-shape filter."

Accordingly, JP '357 no longer anticipates claims 12 and 24. Furthermore, JP '357 does not render claims 12 and 24 unpatentable since it does not suggest using a deep-bed filter for the second stage filtering. To the contrary, JP '357 specifically teaches the use of a cheap, disposable plate-shaped filter for the second stage filtering because of the clogging problem mentioned above.

The deficiencies of JP '357 are not remedied by US '873. US '873 discloses a deep-bed filter which removes inclusions from a metal melt in two steps. In the first step, the filter retains metal-non-wettable inclusions caused by oxide particles. In the second step, the filter retains metal-wettable inclusions arising from grain-refiners, e.g., TiB_2.

See, col. 2, lines 30-34 and 46-48, of US '873. Furthermore, US '873 discloses that adding borides as grain-refiners leads to impurities in the form of clusters up to 30 microns in size. See col. 2, lines 46-56. In view of the teachings of JP '357, however, coarse impurities together with oxide inclusions enhance clogging of the filter by forming a tenacious filter cake. See para. [0013] of JP '357.

Therefore, a person skilled in the art would expect enhanced clogging of the second filter and would not be motivated to use an expensive deep-fed filter as the second filter. Rather, the skilled artisan would be motivated, as in JP '357, to employ a cheap-disposable plate filter for the second filter under these circumstances. Thus, the inventions of claims 12 and 24 would not be obvious to a person of ordinary skill in the art from the teachings of JP '357 when taken together with the teachings of US '873. Since neither US '241 nor US '876 disclose the use of deep-bed filters for filtration of metal melts, it is submitted that the invention set forth in claims 12 and 24 are not rendered unpatentable by any combination of the prior art of record.

As the remaining claims in the application all depend from claim 12, and therefore incorporate each of its limitations by reference, it is submitted that they too are patentable over the prior art of record.

In view of the foregoing, it is believed that the application is now in condition for allowance and a favorable action on the merits is respectfully requested.

Respectfully submitted,
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Attachment: JP '7/207,357 (in Japanese and English)

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EUROPEAN PATENT OFFICE

Patent Abstracts of Japan

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APPLICATION DATE : 17-01-94
APPLICATION NUMBER : 06003285

APPLICANT : KOBE STEEL LTD;

INVENTOR : KITANO TAKAYUKI;

INT.CL. : C22B 21/06

TITLE : FILTRATION METHOD OF MOLTEN AL OR AL ALLOY

ABSTRACT : PURPOSE: To extend the life of an expensive filter by adding a crystal grain size micronizing agent to molten metal in between a first and second stage of a refractory porous filter and filtering.

CONSTITUTION: Molten Al or Al alloy is filtered by using a filter prior to casting. In this case, a refractory porous filter is fitted in two stages. A crystal grain size micronizing agent is added to molten metal in between the first porous filter and second porous filter to filter the molten metal. Also, when each filtration is executed by two stage filters, each molten metal is oxidized to form Al oxide in molten metal and is filtered, at the early stage of filtration, an oxide film is formed on the filtration front side of each filter, a removing efficiency of the inclusions in molten metal is improved. By this method, the adverse impact due to adding a crystal grain micronizing agent is suppressed to a minimum.

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最終頁に続く

(54)【発明の名称】　ＡｌまたはＡｌ合金溶湯の濾過法

(57)【要約】

【構成】　Ａｌ溶湯の濾過に際し、フィルターを２段構成とし、結晶粒微細化剤の添加を第２段濾過の前とする。好ましくは第２段濾過前のＡｌ溶湯を積極酸化し、濾過効率を高める。

【効果】　フィルター、特に第１段濾過で用いる高級フィルターの寿命を延長することができた。

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【特許請求の範囲】

【請求項1】　Alまたは Al合金溶湯を鋳造に先だって濾過するに当たり、耐火性多孔質フィルターを2段に設け、第1段の耐火性多孔質フィルターと第2段の耐火性多孔質フィルターの間で前記溶湯に結晶粒微細化剤を添加して濾過を行うことを特徴とするAlまたはAl合金溶湯の濾過法。

【請求項2】　第1段の耐火性多孔質フィルター及び第2段の耐火性多孔質フィルターによる各濾過を行うに際し、夫々溶湯を酸化することにより該溶湯中にAl酸化物を形成してから濾過を行い、濾過の初期に各フィルターの濾過前面側に酸化物皮膜を形成することにより溶湯中介在物の除去効率を高めて行う請求項1に記載の濾過法。

【請求項3】　第2段の耐火性多孔質フィルターによる濾過を行うに際し、結晶粒微細化剤の添加と同時もしくは前後して該溶湯を酸化することにより該溶湯中にAl酸化物を形成してから濾過を行う請求項2に記載の濾過法。

【請求項4】　第1段の耐火性多孔質フィルターとしてはチューブ状のものを用い、第2段の耐火性多孔質フィルターとしては板状のものを用いる請求項1～3のいずれかに記載の濾過法。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明はAlまたはAl合金溶湯の濾過に際して用いるフィルターの寿命を延長する技術に関するものである。

【0002】

【従来の技術】Alまたは Al合金は、軽量性、加工性、表面美麗性等の特徴を有することから種々の用途に利用されている。しかし不純物元素の混入によって、共晶化合物が粗大化する等の不都合が生じ、強度、靭性、表面処理性等が著しく劣るという問題がある。特に近年は製品の薄肉化、細線化などの要求が厳しくなる傾向にあり、欠陥の発生が無く安定して高品質を示すAlまたはAl合金（以下Alで代表することがある）を提供することが必要となっている。

【0003】Alの製造工程は、①原料の溶解、②溶解炉内での精錬、③炉外での精錬、④鋳造を基本プロセスとして構成されるが、欠陥発生を防止する為には、上記各プロセスの中でも、②と③の精錬プロセスが重要な位置を占める。本発明は③の炉外精錬における最終のステップ、換言すれば④の鋳造の直前に設けられる重要な仕上げステップを構成する濾過技術に関するものである。

【0004】Al製品の欠陥における最大原因の1つは介在物の混入であるが、介在物の由来としては、①の原料に混入して持込まれた酸化物や各種異物、更には②の精錬に当たって溶湯が大気中の酸素と反応することによって形成される酸化物等が考えられる。これら①，②に

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由来する介在物は、実質上回避不可であり、その為上述した様に鋳造直前の最終段階で濾過を行なうことが必須の重要工程となってくるのである。

【0005】しかしながら①，②の工程等で混入してきた介在物の全てを濾過工程で除去することになるので、フィルターの濾過前面側には大量の介在物が捕捉されて堆積し、僅か数チャージで目詰りを起こすという問題がある。ここでフィルターとしては、古くは板状フィルターが用いられていたが、近年はチューブ状フィルターを複数本組合せて濾過面積を拡大しつつチューブの外側から内側に向けて効率的に濾過させるという技術が広く利用されている。しかしチューブ状フィルターは高価であるという欠点があり、しかも前述の如く数チャージの使用によって目詰りが進行し濾過能力が低下する様であると、これを頻繁に廃棄して新品と交換しなければならず、このことがAl製造コストを押上げてしまうということが問題となっている。

【0006】この様なところから、フィルターの寿命延長が課題となり種々検討されている。これらのうち特公昭52－41726号には、フィルターの濾過前面側に付着している無機物質ケーキを溶湯濾過の実施過程中に取除く技術が提案されており、具体的には複数本のチューブ状フィルターを組合わせてなるカートリッジ形式のフィルターセットに対し、その下方から溶湯内へ不活性ガスを吹込んで不活性ガスの微細気泡を吹当て、前記ケーキを強制的にはぎ落して溶湯中に再分散させると共に、溶湯中に溶解しているH_2 ガスを不活性ガス気泡中に拡散させて脱H_2 ガスを平行的に遂行しようとする方法が示されている。

【0007】フィルターの濾過前面側に付着しているケーキ層は極めて強固なものである為、不活性ガスの微細気泡を吹当てる程度では、ケーキの全てを除去し得るに至らず、これに代る新しい手段を開発することが望まれている。一方本発明者等の最近の研究によれば、ケーキ層が強固になって一層剥離され難いものになっていく理由の幾つかが解明された。

【0008】

【発明が解決しようとする課題】従って本発明の目的は、ケーキ層が一層強固になる原因を少しでも解消し、フィルターからのケーキ除去をより容易なものとすることにより、上記の様な公知手法であっても実質的に容易に剥離し得る程度のケーキの形成にとどめることを意図するものである。

【0009】

【課題を解決するための手段】上記目的を達成することのできた本発明は、AlまたはAl合金溶湯を鋳造に先だって濾過するに当たり、耐火性多孔質フィルターを2段に設け、第1段の耐火性多孔質フィルターと第2段の耐火性多孔質フィルターの間で前記溶湯に結晶粒微細化剤を添加して濾過を行うことを基本的構成要旨とするも

のである。尚第１段の耐火性多孔質フィルター及び第２段の耐火性多孔質フィルターによる各濾過を行うに際し、夫々濾過対象の溶湯を酸化することにより該溶湯中にＡｌ酸化物を形成してから濾過を行う様にすれば、濾過の初期に各フィルターの濾過前面側に酸化物皮膜が形成されて見掛け上の目開きが小さくなるので、溶湯中の介在物の除去効率が高まり、従って安定した濾過効果を得ることができる。別の好ましい実施態様によれば、第２段の耐火性多孔質フィルターによる濾過を行うに際し、結晶粒微細化剤の添加と同時もしくは前後して該溶湯を酸化することにより該溶湯中にＡｌ酸化物を形成してから濾過を行うことが可能である。更に別の好ましい実施態様によれば、第１段の耐火性多孔質フィルターとしてはチューブ状のものを用い、第２段の耐火性多孔質フィルターとしては板状のものを用いる方法が推奨される。

【００１０】

【作用】まずＡｌ溶湯濾過におけるフィルターの目詰り状況について説明する。Ａｌ溶湯中に含まれる介在物のうちフイルター除去の対象となるものは、大別して二つに分類される。一つはフィルターメッシュに比べて極めて大きい高融点酸化物であり、主として膜状を呈し、他の一つはフィルターメッシュに比べて極めて小さい高融点酸化物であり、主として微粒状を呈する。濾過に当たっては、始めに上記膜状介在物がフィルターの濾過前面側（以下単に表面ということがある）に沈降してフィルターを構成する骨格部に絡着し短時間の内に目詰りを生じ、見掛上非常に目開きの小さいメッシュフィルターとなる。その為本来ならばフィルターを通過してしまう程の前記微粒状介在物が目開きの小さくなったフィルターに捕捉されることとなる。即ちＡｌ溶湯の濾過は「表面濾過」機構によって行なわれるものである。この様にして１チャージの濾過が終了した後のフィルター表面には、膜状酸化物が絡着し、且つこの膜状酸化物に粒状酸化物が散在的に付着した状態が形成されている。

【００１１】こうして形成されるケーキは、上記形成様式から理解される様にそれ自体かなり強固なものとして成長する素地を有するが、前述した本発明者らの究明に基づく因子が加わって一層強固なケーキが形成されるのである。以下この点について説明する。

【００１２】Ａｌ製品に要求される特性の一つとして強度が挙げられる。この特性を満足する為には結晶粒を微細化することが必要であり、結晶粒微細化剤としてのＴｉやＴｉ－Ｂを、塊状やワイヤ状のＡｌ－Ｔｉもしくは Ａｌ－Ｔｉ－Ｂとして添加することが一般に行われている。従って例えばＡｌスクラップを溶解原料とする場合には、該スクラップ中に配合されているＴｉやＴｉ－Ｂが溶湯中に持込まれることとなるが、このＴｉやＴｉ－Ｂは前記添加時の溶解条件によっては、或は鋳造工程中の冷却速度如何によってはＡｌ製品中にかなり大きい介在物として成長しているものもあり、この様に粗大に成長しているものは当然に、また成長せず微細分散しているものであっても、再溶解後のＡｌ溶湯中ではもはや結晶粒微細化効果を発揮しないこと等も知られている。その為本発明のＡｌスクラップを再溶解して使用することを意図する場合には、結晶粒微細化剤を新たに添加することが必須となっている。しかるにこの結晶粒微細化剤として市中で入手されるのは、前記の様にＡｌ－ＴｉやＡｌ－Ｔｉ－Ｂのワイヤや塊であり、これらを再溶解したＡｌ溶湯中に投入した場合も溶湯中に粗大な不溶介在物として混入することが多いというのが現製造プロセス上の実状である。従ってＡｌ溶湯中には、Ａｌスクラップに由来する粗大なＴｉやＴｉ－Ｂ、更には新たに投入した結晶粒微細化剤中の粗大なＴｉやＴｉ－Ｂが、結晶粒微細化作用を実質的に発揮しない不純物として混入しており、これらの不純物も前記酸化物系介在物と一緒に炉過されなければならないのである。

【００１３】この様な結晶粒微細化剤が混入した状態で濾過を行うと、フィルター表面には前記した如く、膜状介在物の付着に伴う表面濾過機構によって粒状介在物並びに前記粗大なＡｌ－ＴｉやＡｌ－Ｔｉ－Ｂが捕足される。捕足されること自体は濾過の主旨に徹して合目的なことであるが、この様な大小とりまぜた介在物が濾過チャージの進行につれて次々と積層成長していき、遂に濾過不能な程の目詰まりに至るのである。そこで本発明者らはこの様な表面濾過機構の進行状況について自由な角度から検討を行った結果、再溶解後に投入する結晶粒微細化剤がＡｌ溶湯中に十分微細分散されず、一部粗大なまま分散されて結晶粒微細化作用を発揮しないまま、酸化物介在物と共にフィルター表面に捕足されることがケーキの強固さをより一層増大し、フィルター表面からの剥離に対して大きな抵抗になっているのではないかとの示唆を得るに至った。

【００１４】そこで（Ａ）従前通りＡｌ溶湯の濾過工程前に結晶粒微細化剤を添加して濾過した場合と、（Ｂ）該添加を省略して濾過した場合の夫々について実験を行い、濾過を数チャージ行い、夫々フィルター入湯側のヘッド値の変化を比較した。ここでヘッド値とは、フィルターボックスを板状フィルターによって入湯側と出湯側に仕切り、出湯側をオーバーフロー方式として湯面高さを一定とした場合において、入湯側へのＡｌ溶湯注入量を制御して出湯側におけるオーバーフローが定常的に行われる状態における入湯側湯面（高位）と出湯側湯面（低位）の高低差を意味する。従ってフィルター表面のケーキ量が増大して目詰まりが強くなるにつれてヘッド値は増大する。

【００１５】この様な実験系においてヘッド値の変化を比較したところ、（Ａ）の従来方式では１つの濾過チャージが終了した時点のヘッド値と、その次の濾過チャージの最初のヘッド値は殆んど変わっていなかった。即ち

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ある1つの濾過チャージ内では濾過進行に伴ってヘッド値が上昇していくが、前チャージで到達したヘッド値を出発点として次チャージの濾過進行に伴うヘッド値の上昇が単純にプラスされていくことが分かった。従ってヘッド値は上昇一方であり、全体として極めて少ないチャージ数で濾過不能の限界域に至る。一方（B）に示した結晶粒微細化剤非添加の系では、ある1つの濾過チャージ内では濾過進行に伴ってヘッド値が上昇してくことに変わりはないが、前チャージが終了した時点のヘッド値に比べて、その次の濾過チャージ開始時点のヘッド値は有意に低い値になっていることが分かった。これは驚くべき知見であり、前チャージの濾過が終了した後、次チャージを行うまでの待ち時間の間、フィルター保護の目的で保持されていた前チャージの残留溶湯がフィルター表面の付着ケーキを自然剥離し、これによって待ち時間中にヘッド値の低下を招いたものと解釈される。この様な現象は従来全く知られておらず、新たに添加することが常識とされていた結晶粒微細化剤がケーキの剥離を阻害する原因の一つであることを突きとめた功績は頗る大きいものである。

【0016】この様な知見を基礎として、結晶粒微細化剤はフィルターの前段に添加しないとの基本構成に到達したが、Al製品の強度特性を高めるという観点からは結晶粒微細化剤はいずれかの時点で添加されなければならず、該添加に際して混入してくる前記粗大な結晶粒微細化剤は何らかの手段によって除去されなければならない。そこで種々検討を重ねた結果、フィルターを2段構成とし、結晶粒微細化剤は第2段フィルターの前に添加することとした。この様な構成であれば、第1段目のフィルターは前記（A），（B）の対比から分かる様にフィルター寿命が延長されるので、高価なフィルター、例えばチューブ状フィルターを第1段として使用し、安価で使い捨てても良いと思われる板状フィルターを第2段フィルターとして使用する、といった使い分けも可能となる。尚、多段濾過そのものについては例えば特開平2－211937等で知られているが、これは溶湯の層流化を意図するものであり、本発明とは目的、構成共に全く相違するものである。

【0017】一方前記した様な表面濾過機構の説明から理解し得る様に、膜状の酸化物系介在物は第1段目のフィルターで捕足されており、第2段目のフィルターにおける濾過に際しては溶湯中に存在しない。そこで第2段目のフィルター直前でAl溶湯を積極酸化し、膜状介在物を形成して第2段フィルターにおける表面濾過機構の発揮に寄与せしめることは有用な手段と考えられる。その様な積極酸化を行う為の具体的な手段としては、下記の方法が例示される。

【0018】（1）溶湯中に空気を吹込む方法

溶湯内部が空気によって直接酸化されると共に、吹込み空気による溶湯撹拌効果によって溶湯表面が大気酸化を

6

受け、速やかに膜状酸化物が形成されてこれをフィルター表面に付着させることができるので、次チャージ濾過の初期から安定した表面濾過に移行していく。

（2）溶湯中に不活性ガスを吹込む方法

溶湯表面が不活性ガスによる撹拌作用によって大気酸化を受け易くなり、前記空気吹込みよりは若干遅れるが、速やかに膜状酸化物が形成される。

（3）溶湯を機械撹拌または電磁撹拌する方法

上記（2）と実質的に同様の作用効果が発揮されて速やかに膜状酸化物が形成される。

【0019】この様な強制的酸化方法によって溶湯中に形成される膜状酸化物は、種々実験した結果、一般にmm単位～数百μmの大きさであり、微小な粒状酸化物は殆ど生成しないことが確認されている。一方一般に用いられるフィルターの目開きは通常数百μm以下であるから、上記膜状酸化物はフィルター表面を目詰りさせるに十分な大きさであり、短時間の酸化によって目開きの小さなフィルターに改変し、次チャージの濾過に備えることができる。上記した強制的酸化方法によってどの程度の膜状酸化物が生成されるかは、予め溶湯及びフィルターの条件等を変動させて予備試験を行なって承知しておくことが推奨される。

【0020】上記した様なAl溶湯の積極酸化は、第2段フィルターの前だけでなく、必要であれば第1段フィルターの前に実施して表面濾過機能を高めることに寄与せしめることもできる。またAl溶湯の積極酸化を第2段フィルターの前で行う場合には結晶粒微細化剤の添加と同時に、又は相前後して実施すれば良い。

【0021】

【実施例】下記条件でA1原料溶解以降の諸工程を行い、鋳塊を得た。

原料：品種ＪＩＳ　３００４合金，屑配合７０％＋地金（９９．７％Ａｌ）３０％

溶解：溶解炉・重油焚き反射炉（３０ｔ），大気溶解，７４０℃

精練：溶解炉の溶湯中にCl_2ガス，２００Ｎｌ／分×２０分吹込み，除滓

保持：重油焚き反射炉（同上）にて溶湯保持

濾過：第１段　三井金属製チューブ状フィルター（ＨＤ）

１８本組１セットを使用

第２段　神戸製鋼所製アクトサーミックスフィルター

系径２mmφ

大きさ５００mm×５００mm×５０mm

１枚使用

溶湯流れに対して垂直に設置

結晶粒微細化剤の添加（A）＝Ａｌ－Ｔｉ－Ｂワイヤ溶湯中にＴｉ＝０．０１％，Ｂ＝０．００２％となる様に添加

溶湯の積極酸化（B）＝第２段フィルター前の溶湯に対

し、ランス（10mmφを用い、Arガスを5リットル／分，10分間吹込み

実験結果は表1に示す通りである。

【0022】

【表1】

No.	チューブ状フィルターの寿命	板状フィルターの寿命	鋳塊品質	備考	総合判定	(A)，(B) の実施順序
1	1000t	30t／1ch使い捨て	介在物欠陥レベルppm単位以下			第2段フィルター前に(A)→(B)
2	1000t	30t／1ch使い捨て	介在物欠陥レベルppm単位以下		○	第2段フィルター前に(B)→(A)
3	1000t	30t／1ch使い捨て	介在物欠陥レベルppm単位以下		○	第2段フィルター前に(A)，(B)
4	1000t	30t／1ch使い捨て	介在物欠陥レベル100ppm	結晶粒微細化剤の内粗大なものは板状フィルターで捕集困難	×	第2段フィルター前に(A)のみ実施、(B)を省略
5	1000t	－	介在物欠陥レベル200ppm	結晶粒微細化剤の粗大なものがそのまま鋳塊中に存在	×	同上 板状フィルターも省略
6	600t	－	介在物欠陥レベルppm単位以下	チューブ状フィルター寿命短い。但し品質良好	×	(A)を第1段フィルター前で実施、(B)を省略 板状フィルターも省略

【0023】表1に見られる様に2段濾過構成で且つ第2段フィルターの前で結晶粒微細化剤の添加と溶湯の積極酸化を併用したものは最善の結果を与えた。特に第1段濾過に用いたチューブ状フィルターの寿命を約1．5倍延長できた。

【0024】

【発明の効果】本発明は上記の様に構成されているので、結晶粒微細化剤の添加による悪影響を最小に抑えつつ、高価なフィルターの寿命を延長することが可能となった。

フロントページの続き

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Translation of the description of the Japanese laid-open patent application JP-A 7-207357

Title: Filtration method for molten Al or Al alloy

Claims

1. Filtration method for molten Al or Al alloy, characterized in that refractory porous filters are provided in two stages when molten Al or Al alloy is subjected to filtration and that the filtration is performed by adding a grain refiner between the first refractory porous filter and the second refractory porous filter.

2. A filtration method for molten Al or Al alloy according to claim 1, wherein the filtration by the first refractory porous filter and the second refractory porous filter is performed after formation of Al oxide the molten metal by means of oxidation of each molten metal in order to form an oxide film on the filtration front side of each filter at the early stage of filtration.

3. A filtration method for molten Al or Al alloy according to claim 2, wherein the oxidation of said molten metal at the stage of filtration using the second refractory porous filter is carried out simultaneously or before or after the addition of the grain refiner.

4. A filtration method for molten Al or Al alloy according to one of the aforementioned claims, wherein a tube filter is used as first refractory porous filter and a plate filter is used as second refractory porous filter.

Detailed Description of the Invention

0001 Industrial Field of the Invention

The Invention relates to a technique for the extension of the lifetime of filters used for the filtration of molten Al or Al alloys.

0002 Prior Art

Because of their low weight, their processability and the decorative possibilities of their surface Al and Al alloys are used for various applications. The product quality can be damaged by contamination of the material by impurities. The latter can result in the formation of coarse eutectic compounds which have a negative impact on

material properties like strength, rigidity and treatability of the material surface. Particularly in recent years the requirements towards material quality have become tougher with the trend to thickness reduction of Al products and to thinner wires. Therefore, it became necessary to deliver Al and Al alloys (hereafter both referred as Al) with a defect-free stable high quality.

0003

The production of Al can be divided into the following four basic processes:

1) Melting of the raw material,

2) Refining in the smelting furnace,

3) Refining outside of the furnace,

4) Casting.

Thereof especially the refining processes are of great importance as far as the prevention of material defects resulting from impurities is concerned. The present invention is related to the filtration of the Al melt performed as last step of the refining process outside the furnace (process no. 3) or, in other words, as a step directly in advance of the casting (i.e. process no. 4).

0004

The most important reason for defective Al products is the contamination with inclusions. These inclusions can come from two sources, on the one hand they result from oxides and other impurities introduced as raw material, on the other hand they can result form oxides formed during the refining process in smelting furnaces when the molten material reacts with atmospheric oxygen. Both types of inclusions are practically irreversible so that they must be filtered off directly in advance of casting. The filtration of the Al melt therefore is a very important production step.

0005

Since all inclusions that are introduced into the material during the processes no.1 and no. 2 have to be eliminated, great amounts of inclusions are collected in the filtration front side of the filters, this circumstance results in a clogging of the filters already after having treated only a few charges. In older times plate filters were used

as filters but in recent years a technology found wide usage that combines several tube filter elements and thus provides an enlargement of the filter area surface and therefore results in a more effective filtration from outside to inside. A disadvantage of this technology can be seen in the high price of these tube filters. Related to the quick clogging of the filters they have to be disposed and exchanged quite frequently. This results in a disadvantageous increase of the production cost of Al.

0006

Under these circumstances the objective of a prolongation of the filter lifetime has been object of various studies. E.g. in the JP-B 52-41726 a technique is proposed that provides an elimination of the inorganic cake sticking to the filtration front side. This technique is applied to cartridge-type filter sets consisting of a combination of several tube filters. An inert gas is blown into the melt from downside so that fine inert gas bubbles are blown onto the said cake in order to detach it from the filter surface, then it is redispersed into the molten metal. Parallel to this it is tried to eliminate H_2 dissolved in the molten metal by diffusion of it into the inert gas bubbles.

0007

However, since the cake layer is a very tenacious matter a complete removal of the cake cannot be achieved by simply blowing fine inert gas bubbles onto it. Therefore, there is a great demand for a new means that can replace this technique. Recent studies of the inventors revealed that there are several reasons for the build-up of said tenacious cake layer that cannot be detached easily due to its tenacity.

0008 Objective to be solved by the Invention

Under these circumstances it is an objective of the present invention to reduce the influence of factors that cause said solidification of the cake layer and o enable an easier removal of the cake from the filters, i.e. to stop the cake formation at a level at which it can be easily and substantially eliminated by the known method as described above.

0009

The basic characteristics of this invention that could solve aforementioned objective consist in providing refractory porous filters in two stages when molten Al or Al alloy is subjected to filtration and in that said filtration is performed by adding a grain refiner between the first refractory porous filter and the second refractory porous filter. If the filtration by the first refractory porous filter and the second refractory porous filter is performed after formation of Al oxide the molten metal by means of oxidation of each molten metal, an oxide film on the filtration front side of each filter is formed at the early stage of filtration so that a higher elimination ratio for inclusions in the molten material is achieved due to a decreased mesh size. According to a further preferred embodiment of the invention, the oxidation of the molten metal at the stage of filtration using the second refractory porous filter is carried out simultaneously or before or after the addition of the grain refiner. This allows performing the filtration after the formation of Al oxides within the molten metal. According to a further preferred embodiment of the invention a tube filter is used as first refractory porous filter and a plate filter is used as second refractory porous filter.

0010

Functioning of the Invention

In the following at first the clogging state of the filter is discussed. The inclusions in the molten Al that are subjected to filtration can roughly be classified into two groups. The first one consists of oxides with a high melting point that have extremely larger diameters compared to the filter mesh size and are mainly present in a film-shape. The second one is made up of high melting oxides being mainly present as fine particles with diameters which are extremely smaller than the filter mesh size. In the first stage of filtration the film-type inclusions are deposited on the filtration front side (hereafter simply called surface). When these inclusions adhere to the lattice constituting the filter the filter will be clogged in a short time so that it appears soon as a filter with an extremely small mash size. Therefore, even the particulate-type inclusions that ordinarily would have passed the filter are collected by the filter. This means that the filtration of molten Al usually relates to a "surface filtration" mechanism. In this case the filter surface is covered with the film-type oxide adhering onto the surface after the passage of only one charge. In addition, this results in a

state in which particulate oxides are sticking onto these film-type oxides in a dispersed distribution.

0011

It can be easily understood that a cake formed in the discussed manner has a base that will show a growth resulting in a rather firm matter so that a cake will be formed that shows an even stronger tenacity. This is due to reasons that will be discussed in the following and that lead - together with the reasons found by the studies of the inventors and described above - to the high tenacity of the filter cake.

0012

One important characteristic required from Al products is mechanical strength. In order to meet this requirement it is necessary to refine the crystal grain size, therefore it is a common practice to use grain refiners, e.g. Ti or Ti-B. When Al scrap is used as raw material these grain refiners are added as Al-Ti or Al-Ti-B wires or lumps. Being introduced into the Al melt after being added to the Al scrap it can occur that Ti or Ti-B grow to relatively coarse inclusions within the Al products depending on the melting conditions at the time of their addition or the cooling rate during the casting process. It is a known fact that Ti or Ti-B as inclusions loose their grain refining activity in the remolten Al melt. This is naturally the case when they are present as coarse inclusions but also when they show no further growth or when they are fine dispersed. Therefore it is necessary to re-add the grain refiner if it is intended to use remolten Al scrap according to the present invention for Al production. However, the grain refiners found on the market are Al-Ti and Al-Ti-B in the form of wires and lumps and during the manufacturing process these grain refiners often cause contamination in the form of insoluble coarse inclusions when being added to remolten Al melt. Therefore, in addition to coarse Ti and Ti-B inclusions from the Al scrap coarse Ti and Ti-B inclusions as ingredients of the newly added grain refiner are present in the Al melt. Since these impurities have lost their grain refining activity they must be filtered off together with the aforementioned oxide inclusions.

0013

When the molten metal containing the aforementioned grain refiners is subjected to filtration, the aforementioned coarse Al-Ti and Al-Ti-B impurities are collected together with the particulate inclusions due to the aforementioned surface filtration

mechanism that, as described above, appears when film-type inclusions are adhering onto the filter surface. The fact that these are collected itself is in accordance with the objectives of the filtration but the inclusions comprised in the Al melt successively accumulate and grow with the progression of the filtration so that in the end the clogging of the filter reaches an extent at which a further filtration is impossible. As a result of a further study of the progression of the aforementioned surface filtration mechanism from various perspectives the inventors found that the grain refiner that is added to the remolten Al melt is not sufficiently finely dispersed in the melt and partly dispersed in the state of coarse aggregations so that it is collected together with the oxide inclusions on the filter surface without performing its grain refining activity. This fact obviously enhances the tenacity of the filter cake and is very likely to be the reason for the strong resistance of the cake against the attempt to peel it off from the filter surface.

0014

Thereupon the inventors carried out experiments comprising the filtration of several charges, wherein in case A grain refiner was added before the filtration (conventional method) and in case B the filtration was carried out without previous grain refiner addition. These experiments were undertaken in order to compare the variation of the head value at the melt inlet side of the used filters. Head value in this context means the difference in altitude between the melt level at the filter inlet side (high level) and the melt level at the filter outlet side (low level) under a steady overflow at the outlet side controlled by the amount of melt charged to the inlet side (the filter box was divided into a melt inlet side and a melt outlet side by a plate-type filter and the altitude of the melt level is kept constant at the outlet side by overflow). Hence the head value increases with the intensification of the clogging due to the increase of the cake on the filter surface.

0015

The comparison of the head value variation in the described experiment led to the result that in case A (conventional method) the head value at the end of one filtration charge did almost not differ from the head value at the beginning of the next filtration charge. This means that the head value increases within one filtration charge in accordance with the progression of the filtration, but the head value increase resulting from the filtration progression of the following charge (with the head value

reached by the previous charge) is simply just added. Hence the head value can only increase so that the limit area whereat a further filtration becomes impossible is already reached after having processed only a few charges. In case B, i.e. filtration without addition of grain refiner, the head value also increases within one filtration charge in accordance with the progression of the filtration. In this point there is no difference with case A. However, the head value at the beginning of the following charge is significantly lower than the head value at the end of the previous charge. This is a really astonishing new finding and can be interpreted as that the head value decrease observed during the waiting time between the end of the filtration of one charge and the start of the filtration of the next charge is due to an auto-releasing action performed by the residual melt of the previous charge that is kept in the filter for filter protection during said waiting time and obviously releases the sticking cake from the filter surface. The elucidation of the fact that the re-addition of grain refiners hinders the release of the cake provides indeed many merits.

0016

Based on this finding, it one fundamental constitutive element of the present invention that grain refiners are not added in advance of the filtration. However, in order to increase the mechanical strength of Al products it is necessary to add a grain refiner during the manufacturing process and it is further necessary to establish a means to eliminate aforementioned coarse grain refiners that are introduced into the melt by re-addition. As a result of intense studies the present invention is characterized in that a filter is provided at two stages and that the grain refiner is added before the second filter. As one can understand from the comparison between case A and B as described above, this constitution allows a prolongation of the lifetime of the first filter. Hence an expensive filter, e.g. a tube-type filter, is used as filter at the first stage while a cheap and therefore disposable plate-shape filter is used at the second stage. By the way, a filtration at several stages is disclosed e.g. in Japanese laid open patent application no. JP 2-211937 A, but said application intends for a laminar flow of the melt and completely different from the present invention as far as its objectives and constitution is concerned.

0017

As can be understood from the aforementioned description of the surface filtration mechanism the film-type oxide inclusions are collected within the first stage filter so that there are no film-type oxide inclusions in the melt when it is filtered with the second stage filter. Thereupon it is a useful means in order to contribute to the surface filtration mechanism in the second stage filter to form film-type inclusions in the Al melt by subjecting it to an active oxidation directly before the second stage filter. This active oxidation can be carried out e.g. according to the methods described in the following.

0018

(1) Blowing air into the melt

By the air blown into the melt its interior is directly subjected to oxidation. Furthermore the melt surface is oxidized at the atmosphere due to the agitation effect of the air blown into the melt. This allows a rapid formation of film-type oxides. Since these oxides can adhere onto the filter surface, the next charge undergoes a stable surface filtration from the beginning.

(2) Blowing inert gas into the melt

The melt surface is agitated by the inert gas so that the surface can easily be oxidized by the atmosphere. The oxidation happens not as rapid as by blowing air into the melt but nevertheless it results in a rapid formation of film-type oxides.

(3) Mechanical or magnetic agitation of the melt

This method has substantially the same effect like the two previous methods and also results in a rapid formation of film-type oxides.

0019

Investigations revealed that the size of the film-type oxides formed by the aforementioned oxidation methods ranges from millimeters to some hundred µm and that almost no particulate oxides are formed by application of said compulsory oxidation methods. These sizes are sufficient in order to cause a clogging of the filter surfaces since the maximal mash size of usual filters is about some hundred µm. Due

to the rapid oxidation the filter will soon be changed into a small mash size filter and thus can be used for the filtration of the next charge. As far as the properties of the film-type oxides produced by the aforementioned compulsory oxidation methods are concerned it is recommended to investigate them by preliminary tests conducted under various conditions for the melt and the filter.

0020

The aforementioned active oxidation of the Al melt is not restricted on conduction before the second stage filter; if necessary, it can be conducted also before the first stage filter. This will contribute to a higher surface filtration performance. When the active oxidation is carried out before the second stage filter, it can be conducted simultaneously with the grain refiner addition but also before or after the grain refiner addition.

0021

Working Examples

Castings were obtained by carrying out the following processes starting with the melting of the raw material.

Raw material:	alloy JIS 3004 consisting of 70% scrap and 30% ground metal (Al 99.7%)
Melting:	30 t reverberatory furnace fired with heavy oil, atmospheric melting at 740 °C.
Refining:	blowing of Cl_2 gas into the melt (200 N1/min x 20 min.), deslagging
Soaking:	holding of the melt in a reverberatory furnace fired with heavy oil (same as above)
Filtration:	1st stage: tube filter HD ex Mitsui Mining & Smelting (1 unit of 18 filter elements) 2nd stage: "Actothermix Filter" (ex Kobe Steel, diameter: 2 mm, size: 500 mm x 500 mm x 50 mm) provided vertically towards the melt stream

Addition of

grain refiner (A): Al-Ti-B wire (0.01% Ti, 0.002% B in the melt

Oxidation (B): before 2nd stage filter, 10 min blowing of 5 l/min of Ar with a lance (10 mm diameter)

The results are arranged in table 1.

0022

Table 1

No.	lifetime of tube filter	lifetime of plate filter	quality of castings	remarks	general evaluation	order of execution of A and B
1	1000 t	disposed after 30 t/charge	inclusion defects beyond ppm level			A, then B (before 2nd stage filter)
2	1000 t	disposed after 30 t/charge	inclusion defects beyond ppm level		good	B, then A (before 2nd stage filter)
3	1000 t	disposed after 30 t/charge	inclusion defects beyond ppm level		good	simultaneously A and B before 2nd stage filter
4	1000 t	disposed after 30 t/charge	level of inclusion defects: 100 ppm	coarse inclusions from the grain refiner could not be collected in the plate filter	bad	only execution of A (before 2nd stage filter), B omitted
5	1000 t	-	level of inclusion defects: 200 ppm	coarse inclusions from the grain refiner remained unchanged in the castings	bad	only execution of A, plate filter omitted
6	600 t	-	inclusion defects beyond ppm level	short lifetime of the tube filter but good quality	bad	execution of A before 1st stage filter, B and plate filter omitted

0023

As it can be understood from table 1, good results were obtained in the examples wherein the melt was subjected to the filtration on 2 stages and wherein the grain refiner was added before the 2nd stage filter in combination with oxidation of the melt. It is remarkable that the lifetime of the tube filter used as 1st stage filter could be prolonged with the factor 1.5.

0024

Advantages of the invention

The constitution of the present invention as described above allows to keep negative impacts resulting from the addition of grain refiners can be reduced to a minimal level and to prolong the lifetime of expensive filters.